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Laboratory Procedure for Determining Percent Ash and Percent Weight Loss of Solid Wastes on Heating at 600 C

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LABORATORY METHODS FOR DETERMINING PERCENT ASH AND PERCENT WEIGHT LOSS OF SOLID WASTES ON HEATING AT 400 C

A Division of Research and Development
Open-File Report (SW-03-68-17)

U.S. ENVIRONMENTAL PROTECTION AGENCY
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A variety of analytical procedures have been employed for the determination of the volatile solids in solid wastes. Researchers, such as Kaiser, have adapted the ASTM standard procedure for coal to the analysis of refuse and residue. Other investigators, such as Schoenberger and Wiley Spillane, have attained more complete oxidations by utilizing open-crucible techniques.

A modification of the procedure proposed by the American Public Works Association has been used extensively by the Research Services Laboratory Staff for the characterization of refuse, residue, and fly ash. After the observation of the weight lost by each sample, the percent ash and the percent weight loss on heating at 600 C are calculated.

A detailed description of the procedure is presented.

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DISCUSSION

Incineration has been employed since 1874 as a method of solid waste reduction[1]. Although early incinerator designs reflected the desire for recovering released heat for steam production and for eliminating or reducing potentially hazardous materials, recent designs have been influenced by a need for more efficient solid waste reduction and lower costs. Engineers and scientists have used various parameters*, e.g., the percent heat released, and the percent reduction in volume, weight, and volatile solids, to evaluate the reduction efficiency of incinerator designs and plan for the economic disposal of the residue[2,3].

A variety of analytical procedures have been employed for the determination of the volatile solids in solid wastes. Researchers, such as Kaiser[4], have adapted the ASTM standard procedure for coal[5] to the analysis of refuse and residue. The heating of the sample in a closed crucible, as directed in the ASTM technique, prevents, however, the oxidation of elemental carbon and results in a weight loss attributable only to the volatilization of hydrocarbons. Other investigators, such as Schoenberger[6] and Wiley and Spillane[7], have attained more complete oxidations by utilizing

*In this paper the term parameter means a variable or characteristic of interest.

open-crucible techniques.

A modification of the procedure proposed by the American Public Works Association[8] has been used extensively by the Research Services Laboratory Staff for the characterization of refuse, residue, and fly ash. Briefly, the technique involves (1) the introduction of two 2-g samples, each contained in a porcelain crucible, into a cold muffle furnace, (2) the gradual increase in furnace temperature to 600 C, and (3) a 2-hour exposure of the samples to the latter temperature. While the crucibles are in the furnace, the lids are either removed or tilted at an angle, sufficiently large to insure the circulation of air over the samples. After the completion of the exposure of the samples to heat, the crucibles are removed from the furnace, immediately covered with their respective lids, and cooled to room temperature in a desiccator. After the observation of the weight lost by each sample, the percent ash and the percent weight loss on heating at 600 C are calculated*.

A detailed description of the procedure is presented in the following sections. The details of an independent Division of Research and Development study of the applicability of this method for the characterization of solid wastes is reported elsewhere[9].

*Since the observed decrease in sample weight reflects not only the weight lost by the vaporization of volatile materials and the combustion of fixed carbon, but also any weight gained, e.g., by oxidation of metallic components during heating, the term, weight loss on heating, appears more appropriate than the term, volatile solids. The term, ash, denotes, of course, the material remaining in the crucible after heating the sample.

EQUIPMENT

1. Balance, analytical, 0.1 mg readability
2. Boxes, sample storage, seamless aluminum, 3-1/2 inches in diameter, with tight-fitting lids (e.g., Fischer No. 3-485*)
3. Crucibles, porcelain, Coors®, high form, size 2, with lids
4. Desiccator, large, either pyrex or stainless steel cabinet-type
5. Furnace, muffle, with indicating pyrometer and temperature controller (e.g., Lindberg Hevi-Duty Muffle Furnace, Model 51333; with automatic Control Console, Model 59545; platinum, platinum--13 percent rhodium thermocouple; and six extra globar, silicon carbide heating elements)
6. Gloves, asbestos, 11 inches long, provided with separate places for thumb and four fingers
7. Gloves, cotton, unlined (e.g., No. 2200 MUH, Wash-Rite, Inc., 1410 Cornell, Indianapolis, Indiana)
8. Ink, ceramic, marking, Coors®, black
9. Mats, board, asbestos, 1/8 by 12 by 12 inches
10. Oven, drying, forced-air (or mechanically convected), capable of maintaining temperatures up to 110 C
11. Pen, steel, for applying ceramic ink
12. Potentiometer, direct reading, with chromel-alumel thermocouple and scale, capable of reading temperatures from 0 to 1000 C (e.g., West Pyrotest, Model 9B, West Instrument Corp., Schiller Park, Illinois)
13. Spatula (e.g., Scoopula, Fischer No. 14-357)
14. Tongs, crucible, nickel-plated, 20 inches long (e.g., Fischer No. 15-208)

*Mention of commercial products does not imply endorsement by the U.S. Government.

REAGENTS

1. Inert standard (e.g., ground McDanel, high temperature porcelain combustion tube fragments)
2. Combustible standard (e.g., A.C.S. sucrose or benzoic acid)

SAMPLE PREPARATION

A solid waste sample must undergo physical preparation before its characterization is initiated in the laboratory. First, it must be dried to constant weight preferably in a forced-air (or mechanically convected) oven. A temperature of 70 to 75 C should be utilized to dry municipal refuse (or compost); incinerator residue and fly ash may be dried at 100 to 105 C. The particle size of the dried sample should be reduced to 2 mm or less using a hammermill, pulverizer, or laboratory mill. To ensure sample homogeneity the ground samples should be thoroughly mixed. Finally, since samples may absorb moisture during the grinding and mixing processes, they should be redried for three hours at the previously specified temperatures and then stored in a desiccator until the analyses are completed.

SAFETY PRECAUTIONS

The following suggestions apply to the analysis of samples:

1. Personal burns can be prevented by introducing the crucibles into a cool furnace and employing asbestos gloves and long tongs while handling the hot crucibles and lids.
2. Bench top damage can be prevented by using asbestos mats to support the hot crucibles and lids before their introduction into the desic-

cator.

3. Breakage of desiccator glass can be prevented by (1) cooling the hot covered crucibles a few minutes before inserting them in the desiccator and (2) avoiding any direct contact of warm crucibles with the glass.

PROCEDURE

The determination of the percent ash and the percent weight loss of each blank, standard, or solid waste sample on heating at 600 C should be performed in duplicate as follows:

Procedure	Comments
1. Transfer a numbered, clean, dry crucible and lid from the desiccator to the pan of an analytical balance.	1. (a) Using a steel pen, apply a Coors® porcelain ink number to the exterior surface of each crucible and lid. Fire the dried ink either with a gas flame or by heating in a muffle furnace at 600 C for 1 hour. (The latter technique also ensures low blank values for new crucibles and lids.) (b) Clean each crucible and lid in warm detergent with a non-metallic brush; rinse first with tap water, then

	with distilled water; dry at 105 C for 1 to 2 hours; assemble; and then cool in a desiccator until needed. (c) In steps 1 through 6 wear cotton gloves to avoid fingerprinting the crucible and lid.
2. Weigh the crucible and lid to the nearest 0.0001 g.	
3. Add approximately 2 g of the prepared sample.	3. Do not add sample to the "blank."
4. Weigh the crucible, lid, and sample to the nearest 0.0001 g.	
5. Transfer the covered crucible to a cool muffle furnace.	5. (a) Use a tray or stack of asbestos mats to transport a number of crucibles simultaneously. (b) WARNING: Introduction of samples into a hot furnace may result in their sudden ignition and loss, and in burns to the analyst. (c) Space the crucibles to permit air circulation around each. (Nine crucibles can be arranged in five rows in the Lindberg furnace.)

6. Carefully tilt each crucible lid at an angle sufficiently large to insure the circulation of air over the sample.

6. Some analysts remove the crucible lids from samples which do not sputter on ignition. Care must then be excerised to return each lid to the proper crucible after the heating is completed.

7. Gradually heat the muffle furnace to 600 C.

7. (a) If using the Lindberg furnace, dial in the desired setting on the digital set point after turning the power and control switches to the "on" position.

(b) It has been observed that higher digital settings are required to achieve 600 C as the elements of the Lindberg furnace age. It is therefore recommended that the Lindberg furnace temperature be monitored once a week with an independent potentiometer and a chromel-alumel thermocouple. The latter may be introduced into the furnace through the small space surrounding the

	door. (c) A 30-minute period is required to heat the Lindberg furnace to 600 C.
8. Maintain the furnace temperature at 600 C for 2 hours.	
9. Then turn off the furnace and immediately transfer first a lid, then its corresponding crucible to a stack of at least three asbestos mats. Recover the crucible immediately.	9. (a) In steps 9 and 10 use an asbestos glove and pair of tongs while handling the hot crucibles. (b) Do not place the hot crucibles or lids directly on table tops or metal trays.
10. Allow the covered crucibles to cool for 3 to 5 minutes, then transfer to a desiccator.	10. Do not permit the hot crucibles to touch the glass of the desiccator.
11. After the crucible, lid, and sample have cooled to room temperature, weigh them to the nearest 0.0001 g.	11. (a) A hot crucible, lid, and and sample usually cool to room temperature in 1 to 2 hours. (b) Wear a cotton glove to avoid finger-printing the crucible and lid during their transfer to the balance.
12. Calculate the initial and final weights, the weight loss on heating, the percent ash, and the percent weight loss on	12. (a) See the section on Calculations. (b) The means of the two determinations of the percent

heating of each sample.

ash and the percent weight loss on heating are usually reported.

STANDARDIZATION

Before initiating the characterization of solid waste samples, the analyst should evaluate his technique by determining the percent ash or percent weight loss on heating of a combustible standard (e.g., benzoic acid or sucrose), an inert standard (e.g., ground McDanel high temperature combustion tube fragments), and three mixtures (e.g., 3:1, 1:1, and 1:3 parts by weight) of a combustible and an inert standard. (See the discussion of accuracy in the section on Method Evaluation.)

Initially and periodically thereafter the analyst should also evaluate the applicability of his technique for cleaning and drying the crucibles and lids. The weight change on heating of a properly cleaned and dried, but empty, covered crucible (or "blank") should not exceed 0.0004 g.

CALCULATIONS

The initial and final sample weights, the weight loss on heating (WLOH), the percent ash, and the percent WLOH may be calculated as follows:

The initial sample weight (g) = B - A

The final sample weight (g) = C - A

The weight loss of the sample on heating (g) = B - C

$$\% \text{ Ash} = \frac{100\ (C - A)}{(B - A)}$$

$$\% \text{ WLOH} = \frac{100\ (B - C)}{(B - A)}$$

$$= 100 - \% \text{ Ash}$$

Where A = the initial weight of the crucible and lid (g)

B = the initial weight of the crucible, lid, and sample (g)

C = the final weight of the crucible, lid, and sample (g)

METHOD EVALUATION

The mean observed percents WLOH of benzoic acid, sucrose, and mixtures of sucrose and McDanel combustion tube fragments were always ≥99.92 percent of the theoretical percent WLOH. (Table 1). Although the mean observed percent WLOH of the 3:1 (parts by weight) mixture of sucrose and combustion tube fragments was 100.45 percent of the theoretical percent WLOH, no weight loss was ever observed when the combustion tube fragments were heated alone.

TABLE 1

ACCURACY OF THE METHOD

Sample		Theoretical % WLOH (T)	Mean observed % WLOH (M)	(100 M/T)
Type	Identity			
Combustible standards	Benzoic acid	100	100.00	100.00
	Sucrose	100	100.00	100.00
	Sucrose-McDanel combustion tube, parts by weight,			
	3:1	75	75.34	100.45
	1:1	50	49.97	99.94
	1:3	25	24.98	99.92
Inert standard	McDanel combustion tube	0	0.00	-

The reproducibility of this procedure may be determined by calculating the standard deviations of the determinations, the standard errors of the means of the determinations, and the coefficients of variation. A review of our observations and calculations (Table 2) indicates that the standard deviations of the determinations of the percents WLOH of the Delaware County, Pennsylvania solid waste samples and the standard errors of the corresponding mean percents WLOH often exceeded and varied more than those of the standard samples, but the coefficients of variation were always less than 0.05. The nature or composition of the solid waste samples and the method of sample preparation may, of course, influence the reproducibility.

Although sufficiently accurate and precise determinations of the percent ash and the percent weight loss of solid wastes on heating at 600 C may be obtained with this procedure in 4 hours, the applicability of the two defined parameters may be limited. Division of Research and Development research has recently demonstrated that some residue and fly ash samples contain significant quantities of carbonate[10]. Since the percent WLOH of Na_2CO_3 and $CaCO_3$ on heating at 600 C is very low, even in the presence of benzoic acid,[11] engineers and scientists may find it advisable to use percent WLOH data collected at 900 C to evaluate the reduction efficiency of some incinerators.

TABLE 2

REPRODUCIBILITY OF THE METHOD

Sample indentity	RSL no.	Mean observed % WLOH (M)	Standard deviation of the determinations (S)	Standard error of the mean ($S/\sqrt{2}$)	Coefficient of variation (S/M)
Combustible standards:					
Benzoic acid		100	0.00	0.00	0.00
Sucrose		100	0.00	0.00	0.00
Sucrose-McDanel combustion tube, parts by weight 3:1		75.34	0.06	0.04	0.00
1:1		49.97	0.14	0.10	0.00
1:3		24.98	0.01	0.01	0.00
Inert standard:					
McDanel combustion tube		0.00	0.00	0.00	-
Solid wastes:*					
Refuse	99	87.58	0.08	0.06	0.00
	100	89.03	0.10	0.07	0.00
	101	86.08	0.06	0.04	0.00
	102	86.76	1.01	0.71	0.01
	103	91.38	0.37	0.26	0.00
Residue, combustible fraction†	105	52.04	0.52	0.37	0.01
	107	56.48	0.90	0.64	0.02
	109	62.96	0.65	0.46	0.01
	111	49.30	0.22	0.16	0.00
	113	61.80	2.21	1.56	0.04
Residue, fines fraction‡	104	15.85	0.17	0.12	0.01
	106	12.63	0.37	0.26	0.03
	108	12.92	0.06	0.04	0.00
	110	11.82	0.16	0.11	0.01
	112	25.63	0.47	0.33	0.02
	112a	49.78	0.25	0.18	0.01
Fly Ash	90	1.80	0.02	0.01	0.01
	91	5.06	0.00	0.00	0.00
	92	4.51	0.04	0.03	0.01
	93	3.81	0.00	0.00	0.00
	94	1.78	0.03	0.03	0.02
	96	2.75	0.01	0.01	0.00
	97	4.58	0.02	0.01	0.00

*These samples were collected at Incinerator No. 3, Delaware County, Pennsylvania.

†The combustible fraction includes most of the residue that can be visually identified as containing food, paper, plastics, rubber, leather, wood, textiles, or garden wastes.

‡The fines fraction includes all the unidentified and some of the identifiable residue particles that pass through a 0.5 inch wire mesh sieve.

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